Field Survey of Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans (PCCDD/Fs) in Sewage Biosolids Treated Ontario Agricultural Soils

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Preface

The public has expressed concern that the concentrations of two organic contaminants, namely polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs), will increase in agricultural soils which receive sewage biosolids, and pose significant risk to human and animal health. The intent of the field survey described herein was to address this concern. The field survey looked at the concentrations of PCDDs and PCDFs in a limited number sewage biosolids treated soils and selected sewage biosolids utilized on agricultural land, and compared the concentrations found in those media to Ontario concentration limits, and also to the findings reported in other studies.

This survey is the second of its type carried out in Ontario to monitor concentrations of parameters of concern in agricultural soils which receive sewage biosolids. The first field survey looked at soil pH, phosphorus and heavy metal levels. The report for that survey is entitled *Analytical Results, Findings and Recommendations of the 1995 OMAF Sewage Biosolids Field Survey* (OMAF, 1995).

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The combined efforts of many people were needed in order to complete the study and this report. The principle contributors were the authors, but the efforts of the following individuals are gratefully acknowledged.

Laboratory analysis of the soils and sewage biosolids for concentrations of PCDDs and PCDFs was carried out by staff of the Dioxin/Furan Unit, Laboratory Services Branch, of the Ministry of Environment and Energy, Etobicoke. The authors would particularly like to thank K. MacPherson and V. Khurana, for their analytical contributions, and E. Reiner for his supervisory contributions. The contributions made by these staff, and the timely delivery of the analytical results, were integral to the study and greatly appreciated by the authors.

The following people assisted with sampling of the sewage biosolids: D. Pellerin and J. Furgal, Regional Municipality of Niagara; M. Crone, Ontario Clean Water Agency (OCWA), Owen Sound; G. Eagles, OCWA, Southampton; B. Rouw, Town of Hanover; A. Lapinski, Regional Municipality of Durham; and M. Janiec and C. Roland, Terratec Environmental Ltd., Oakville. The last two individuals assisted with sampling at sewage biosolids storage facilities in the Regional Municipalities of Halton and Waterloo. The contributions of all are acknowledged with special thanks.

The figures included in the report were generated using computer graphic programs, and were produced by D. Rouleau, Resources and Planning Branch, Ministry of Agriculture and Food, Guelph.

The contributions of the farmer co-operators were also critical for successful completion of this study. This report would not have been possible without their willing co-operation and assistance.

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Field Survey of Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans (PCDD/Fs) in Sewage Biosolids Treated Ontario Agricultural Soils

Executive Summary

The criteria and standards in Ontario for the utilization of sewage biosolids on agricultural land are outlined in the publication entitled ***Guidelines for the Utilization of Biosolids and Other Wastes on Agricultural Land*** (OMEE and OMAF, 1996). They have been developed to facilitate the recycling and use of sewage biosolids on agricultural land, while protecting the quality of food, the health of consumers and livestock, and the quality of the environment.

In 1995, the Ontario Ministry of Agriculture and Food (OMAF) conducted a field survey to study pH, phosphorus and heavy metal levels in soils which had received two or more applications of sewage biosolids. The analytical results and findings of that survey are outlined in the report entitled *Analytical Results, Findings and Recommendations of the 1995 OMAF Sewage Biosolids Field Survey* (OMAF, 1995).

In 1996, further monitoring efforts were carried out in regard to the sewage biosolids land application program in Ontario. The Biosolids Utilization Committee (BUC) endorsed a proposal to carry out a limited field survey to study the concentrations of polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs) in: 1) agricultural soils which had received long-term, multiple applications of sewage biosolids; and 2) sewage biosolids utilized on agricultural land. This report presents the analytical results, findings and conclusions of that field survey.

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PCDDs and PCDFs (PCDD/Fs) are organic chemical compounds which occur naturally as by-products of combustion of organic materials (i.e. during forest fires). Therefore, they can never be totally eliminated from the environment. PCDD/Fs are also toxicants which, if they occur above acceptable levels in environmental media, may pose a risk to human and animal health. PCCD/Fs can occur in sewage biosolids, and the public has expressed concern that the concentrations will increase in agricultural soils which receive sewage biosolids, and pose significant risk to human and animal health.

Since PCDD/Fs are toxicants, it is important that the concentrations of PCDD/Fs in sewage biosolids utilized on agricultural soils, and the concentrations in the receiving soils, not exceed concentration limits which are: 1) specified in relevant Ontario guidelines; or 2) used by the Ontario Ministry of the Environment (OME) in the waste management approval process set out under Part V of the ***Environmental Protection Act*** (R.S.O., 1990). They are as follows:

1) the interim maximum concentration of PCDD/Fs allowed in waste materials applied on agricultural land is **100 ng/kg I-TEQ** (International Toxicity Equivalents) (Pers. Comm., 1997) (Note: 100 ng/kg I-TEQ = 100 ppt I-TEQ);
2) the generic soil remediation PCDD/F concentration for agricultural land use is **10 ng/kg I-TEQ**, as specified in the ***Guideline for Use at Contaminated Sites in Ontario*** (OMEE, 1997). This criterion, however, can not be used for sites defined as sensitive in the guideline, without a site specific risk assessment that demonstrates its acceptability for that site; and
3) the background soil remediation PCDD/F concentration for all land uses is **7 ng/kg I-TEQ**, as specified in the ***Guideline for Use at Contaminated Sites in Ontario*** (OMEE, 1997). This value can be used as a remediation criterion for any site.

Further to 2) and 3), the 10 ng/kg I-TEQ value is an effects based value and has most often been used by the OME as the maximum concentration of PCDD/Fs allowed in agricultural soils which receive waste materials containing PCDD/Fs. The background 7 ng/kg I-TEQ value, however, could also be used if deemed necessary (i.e. sensitive site). Therefore, it was included in this study for comparison purposes.

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Purpose and Objectives

The main purpose of the 1996 field survey was to study the concentrations of polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs) in a limited number of agricultural soils which had received long-term, multiple applications of sewage biosolids. A secondary purpose was to study these parameters in selected sewage biosolids utilized on Ontario agricultural land. Numbers of soil and sewage biosolid samples were limited by the high cost of PCDD/F analysis, estimated at $1,000.00 per sample. Specific objectives of the study were:

1) to determine the concentrations of PCDDs and PCDFs (PCDD/Fs) in Ontario agricultural soils which had received three applications of sewage biosolids;
2) to determine the concentrations of PCDD/Fs in sewage biosolids utilized on Ontario agricultural land, specifically those produced by water pollution control plants which had supplied sewage biosolids to the treated soils; and
3) to compare the measured concentrations of PCDD/Fs in the treated soils, and in the sewage biosolids, to: a) the concentration limits specified in relevant Ontario guidelines, or used by the OME; and b) the concentrations reported in other studies.

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Field Survey

Ten of the paired sites included in the 1995 OMAF field survey were selected and sampled again in 1996. Each site consisted of a "treated" plot located in an agricultural field which had received three applications of sewage biosolids, and a "non-treated" plot located in an adjacent or nearby agricultural field comprising soils with similar physical characteristics, which had not received sewage biosolids. There were a total of 14 farmer co-operators.

Soil samples (0-15 cm depth) were taken at varying intervals along three transects located in each of the treated and non-treated plots. At least 25 samples were taken along the transects, and combined to obtain a composite sample for each plot. The composite sample was then thoroughly mixed, and a portion taken for analysis. A total of 10 treated plots and the same number of non-treated plots were sampled.

Sewage biosolid samples were taken at 7 water pollution control plants (WPCPs) or regional storage facilities which had supplied all, or a portion of, the sewage biosolids applied to the treated soils. Five samples taken at different times were combined into a composite sample for 6 of the WPCPs or storage facilities. Each composite sample was thoroughly mixed, and a portion was taken for analysis. Five samples also taken at different times from the Southampton WPCP were analyzed individually for PCDD/Fs.

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Findings and Conclusions

Two different conventions were used to derive overall toxicity values for the soils and sewage biosolids included in the study. The present international convention (U.S. EPA, 1989) was used to calculate values expressed as International Toxicity Equivalents (I-TEQs), and an earlier international convention was used to calculate values expressed as Homologue Toxicity Equivalents (H-TEQs). The latter values were required for comparison with Ontario background concentrations in soils at uncontaminated rural parkland sites (OMEE, 1994).

The mean concentration of PCDD/Fs in the treated soils was 1.9 ng/kg I-TEQ, and the maximum concentration for these soils was 3.5 ng/kg I-TEQ. These concentrations are well below the generic soil remediation concentration of 10 ng/kg I-TEQ for agricultural land use (OMEE, 1997). They are also well below the background soil concentration of 7 ng/kg I-TEQ (OMEE, 1997), which is the upper limit of Ontario background concentrations in soils at uncontaminated rural parkland sites (OMEE, 1994).

Statistical analysis of the analytical results indicated that there were no significant differences in PCDD/F concentrations between the treated and non-treated soils. Thus, there is no evidence from this study that three applications of sewage biosolids in accordance with the Ontario guidelines (OMEE and OMAF, 1996), increased the concentrations of PCDD/Fs in the receiving soils.

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The 2,3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD) congener is of greatest concern from a toxicity perspective. It was not detected in any of the twenty soils (treated and non-treated), nor in the seven sewage biosolids included in the study.

Using the H-TEQ convention for deriving overall toxicity values, the mean concentration of PCDD/Fs for the treated soils was 1.2 ng/kg H-TEQ, and for the non-treated soils it was 1.3 ng/kg H-TEQ. Both values are slightly lower than the mean value of 1.7 ng/kg H-TEQ reported in the Ontario background database (OMEE, 1994). Moreover, the highest value of 4.1 ng/kg H-TEQ determined for the non-treated soils at the Oakville #2 site, is slightly lower than the 98th percentile value of 4.8 ng/kg H-TEQ also reported in the background database.
Thus, the concentrations of PCDD/Fs in both the treated and non-treated soils are within the normal range of Ontario background soil concentrations (OMEE, 1994).

The mean concentration of PCDD/Fs in the sewage biosolids was 10.8 ng/kg I-TEQ. Concentrations ranged from 5.9 ng/kg I-TEQ in the Whitby sewage biosolids, to 17.8 ng/kg I-TEQ in the Halton sewage biosolids. These values are well below the interim maximum concentration of 100 ng/kg I-TEQ currently allowed in waste materials utilized on agricultural land (Pers. Comm., 1997). The mean value of 10.8 ng/kg I-TEQ for the sewage biosolids also compares favourably with values reported by Webber and Nichols (1995), and Webber and Bedford (1996), who examined concentrations of organic chemical compounds in 16 Canadian sewage biosolids, including 7 from Ontario. They found that the mean concentrations of PCDD/Fs in the sewage biosolids varied from 4 ng/kg I-TEQ to 120 ng/kg I-TEQ. The mean concentration for the 16 sewage biosolids examined was 30 ng/kg I-TEQ, and the mean for the 7 Ontario sewage biosolids was 10 ng/kg I-TEQ.

Thus, based on the findings of this limited study, it is concluded that the application of sewage biosolids to agricultural soils, in accordance with the sewage biosolids guidelines (OMEE and OMAF, 1996), has not resulted in increased PCDD/F risk to human and animal health.

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1.0 Introduction

1.1 Background

Sewage biosolids are the nutrient rich black residues remaining after municipal wastewater treatment at a water pollution control plant. They are a valuable agricultural fertilizer because they contain nitrogen, phosphorus, some trace minerals, and organic matter, which are all beneficial to the soil. The utilization of sewage biosolids on agricultural land can benefit farmers, municipalities and society when carried out in accordance with sound utilization guidelines.

The criteria and standards in Ontario, which must be adhered to in order to utilize sewage biosolids on agricultural land, are outlined in the publication entitled ***Guidelines for the Utilization of Biosolids and Other Wastes on Agricultural Land*** (OMEE and OMAF, 1996). The guidelines supplement the ***General - Waste Management Regulation (Regulation 347), made under Part V of the Environmental Protection Act*** (R.S.O., 1990). They have been developed to facilitate the recycling and use of sewage biosolids on agricultural land, while protecting the quality of food, the health of consumers and livestock, and the quality of the environment. Hereafter, they are referred to as the "sewage biosolids guidelines".

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Members of the public have expressed concerns in regard to the utilization of sewage biosolids on agricultural land. The concern relevant to this study is whether the application of sewage biosolids increases soil levels of polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs), and whether the resulting levels pose an increased risk to human and animal health.

PCDDs and PCDFs (PCDD/Fs) are chlorinated tricyclic aromatic hydrocarbons with similar molecular structures (Figure 1) and physical and chemical properties. They occur in a wide variety of environmental media and have caused great concern in the general public as well as intense interest in the scientific community. Much of the public concern revolves around characterization of these compounds as among the most potent "man-made" toxicants. While this characterization is warranted, it must be balanced by the knowledge that PCDD/Fs occur naturally in soils as by-products of combustion of organic materials (i.e. during forest fires). Therefore, they can never be totally eliminated from the environment. PCDD/Fs can also occur in sewage biosolids. However, the concentrations of PCDD/Fs in sewage biosolids are roughly the same as those found in vacuum cleaner dust, and are considerably lower than in house furnace filter dusts (Berry et al., 1993). Nevertheless, PCDD/Fs are toxicants. Therefore, it is important that the concentrations of PCDD/Fs in sewage biosolids applied on agricultural land, and the concentrations in the receiving soils, not exceed acceptable levels.

The most widely studied of the PCDD/Fs is the 2,3,7,8,-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD) congener. It often is called simply "dioxin", and represents the reference compound for this class of compounds. Dioxin-like compounds are the subset of this class of compounds which are generally agreed to produce dioxin-like toxicity. They include all of the compounds with chlorines substituted in the 2,3,7,8-positions. In order to determine the overall toxicity of these compounds, each is assigned an individual International Toxicity Equivalence Factor (I-TEF) value as defined by international convention (U.S. EPA, 1989). Each I-TEF value is an estimate of the toxicity of that compound relative to the toxicity of the 2,3,7,8-TCDD congener, which is assigned a value of 1.0.

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The sewage biosolids guidelines (OMEE and OMAF, 1996) do not specify the maximum concentrations of PCDD/Fs allowed in: 1) sewage biosolids utilized on agricultural land; and 2) agricultural soils which receive applications of sewage biosolids. While this has lead to public concern and anxiety, concentration limits are either specified in relevant Ontario guidelines, or are used by the Ontario Ministry of Environment (OME) in the waste management approval process set out under Part V of the Environmental Protection Act (R.S.O., 1990).
They are as follows:

1) the interim maximum PCDD/F concentration allowed in waste materials applied on agricultural land is **100 ng/kg I-TEQ** (International Toxicity Equivalents) (Pers. Comm., 1997) (Note: 100 ng/kg I-TEQ = 100 ppt I-TEQ);
2) the generic soil remediation PCDD/F concentration for agricultural land use is **10 ng/kg I-TEQ**, as specified in the ***Guideline for Use at Contaminated Sites in Ontario*** (OMEE, 1997). This criterion, however, can not be used for sites defined as sensitive in the guideline, without a site specific risk assessment that demonstrates its acceptability for that site; and
3) the background soil remediation PCDD/F concentration for all land uses is **7 ng/kg I-TEQ**, as specified in the ***Guideline for Use at Contaminated Sites in Ontario*** (OMEE, 1997). This value can be used as a remediation criterion for any site

Further to 2) and 3), the 10 ng/kg I-TEQ value is an effects based value and has most often been used by the OME as the maximum concentration of PCDD/Fs allowed in agricultural soils which receive waste materials containing PCDD/Fs. The background 7 ng/kg I-TEQ value, however, could also be used if deemed necessary (i.e. sensitive site). Therefore, it was included in this study for comparison purposes.

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In 1995, the Ontario Ministry of Agriculture and Food (OMAF) conducted a sewage biosolids field survey to study pH, phosphorus and heavy metal concentrations in 36 agricultural soils which had received two or more applications of sewage biosolids. The report for that study is entitled *Analytical Results, Findings and Recommendations of the 1995 OMAF Sewage Biosolids Field Survey* (OMAF, 1995).

In 1996, further monitoring efforts were carried out in regard to the sewage biosolids land application program in Ontario. The Biosolids Utilization Committee (BUC) endorsed a proposal to carry out a field survey of PCDD/F concentrations in: 1) agricultural soils which had received long-term, multiple applications of sewage biosolids; and 2) sewage biosolids utilized on agricultural land. The analytical results, findings and conclusions of that field survey are presented in this report.

1.2 Purpose and Objectives

The main purpose of the 1996 field survey was to study the concentrations of polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs) in a limited number of agricultural soils which had received long-term, multiple applications of sewage biosolids. A secondary purpose was to study these parameters in selected sewage biosolids utilized on Ontario agricultural land. Specific objectives of the study were:

1) to determine the concentrations of PCDDs and PCDFs (PCDD/Fs) in Ontario agricultural soils which had received three applications of sewage biosolids;
2) to determine the concentrations of PCDD/Fs in sewage biosolids utilized on Ontario agricultural land, specifically those produced by water pollution control plants which had supplied sewage biosolids to the treated soils; and
3) to compare the measured concentrations of PCDD/Fs in the treated soils, and in the sewage biosolids, to: a) the concentration limits specified in relevant Ontario guidelines, or used by the OME; and b) the concentrations reported in other studies.

Further to 1) above, most agricultural soils in Ontario which have received sewage biosolids, have not received more than three applications under three separate OME Part V approvals. Treated soils which had received three applications were therefore targeted for inclusion in the study.

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1.3 Constraints and Limitations

The study was constrained by the high cost of PCDD/F analysis, estimated at $1,000.00 per sample. This limited the numbers of soil and sewage biosolids samples. Only the minimum number of paired sites needed for statistical analysis of the data were studied. Further, only a single sample, rather than replicate samples, was taken for: 1) each of the treated and non-treated soils comprising the paired sites (20 samples); and 2) each of the sewage biosolids (6 samples), with the exception of the Southampton sewage biosolids (5 samples). The five samples taken at the Southampton water pollution control plant were analyzed individually for PCDD/F concentrations.

The paired sites were good candidates for the study. However, replicate sampling of a greater number of paired sites would have been preferred to allow more comprehensive statistical analysis, and to improve the confidence level of the findings.

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2.0 Field Survey

2.1 General

In the spring of 1996, a partnership was formed consisting of staff and resources of the OMAF, OME, and Water Technology International Corporation (WTI). The main intent of this partnership was to develop and carry out a field survey of PCDD/F concentrations in sewage biosolids treated agricultural soils. Site selection began and was completed in June. The 10 paired sites selected for the study had been previously located as part of the 1995 OMAF sewage biosolids field survey. Soil sampling began in July and was completed in August, and sewage biosolids sampling began in August and was completed in December.

Laboratory analysis of the soil and sewage biosolids samples was carried out by staff of the Dioxin/Furan Unit, Laboratory Services Branch, of the Ministry of Environment and Energy, Etobicoke. Analysis of all samples was completed by February, 1997.

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2.2 Study Methods

2.2.1 Site Selection

Ten of the paired sites included in the 1995 OMAF sewage biosolids field survey (OMAF, 1995) were selected for the study. The following criteria were used to select the sites:

- the treated soil at each paired site had received three applications of sewage biosolids, as per the conditions specified in three separate OME Part V approvals (Certificates of Approval); and
- the treated and non-treated soils at each paired site had the least variability in physical characteristics, of those comprising the paired sites included in the 1995 OMAF field survey.

The farmers who owned or managed the fields comprising the 10 paired sites were again approached and asked to participate in the study. Fourteen farmers co-operated in the study.

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2.2.2 Sampling and Data Collection

Small areas or plots within each treated and non-treated field were identified and sampled. The size of the sampled plots ranged from approximately 2 to 4 hectares. Before each plot was sampled, all equipment was thoroughly cleaned following standard OME procedures to remove organic contaminants.

Soil samples (0-15 cm depth) were taken along three transects in each of the plots. A minimum of 25 samples were taken along the transects by soil probe (2 cm diameter), and the samples were combined into a composite sample. The sampling interval along the transects varied depending on the length of the transect in each plot. Sampling intervals were generally greater than 10 m. The composite sample was mixed thoroughly, and a 250 mL portion taken for analysis. The sample portion was placed into a widemouth amber glass jar with an aluminum foil-lined lid, which had been thoroughly cleaned to remove organic contaminants. The jar was placed in a portable cooler containing ice, and later transferred first to a refrigerator for temporary storage, and then to the laboratory.

Seven sewage biosolids were also sampled and analyzed for PCDD/Fs. Four of the sewage biosolids were from either a water pollution control plant (WPCP) or regional storage facility which had supplied the biosolids applied to 5 of the treated soils. Three of the sewage biosolids were from a WPCP or regional storage facility which had supplied a portion of the biosolids applied to the other 5 treated soils. The latter three sewage biosolids were those obtained from the Grimsby WPCP, Whitby WPCP, and Halton regional storage facility (blend of sewage biosolids).

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Five 250 mL samples of sewage biosolids were taken at different times at each WPCP or regional storage facility. Each 250 mL sample was placed in a widemouth amber glass jar with an aluminum foil-lined lid. Except for Southampton, biosolids samples from the WPCPs and the Halton and Kitchener regional storage facilities, were emptied into a stainless steel bowl which had been cleaned following standard OME procedures. Each composite sample was then thoroughly mixed, and a 250 mL portion taken for analysis. The portion was placed in a widemouth amber glass jar and kept in a refrigerator for temporary storage. When the sampling was completed, the jars were transported on-ice to the laboratory.

The five samples taken at the Southampton WPCP were analyzed individually for concentrations of PCDD/Fs and mean concentrations were calculated for homologues and individual congeners. Those results were then used to derive a mean total toxicity value. The number of samples collected by region/county are given in Table 1.

2.2.3 Sample Analysis

The soil and sewage biosolids samples were air dried and ground prior to analysis. Known quantities of ^{13}C-labelled PCDD and PCDF surrogates were added to each sample to serve as Internal Quantitation Standards. PCDD/Fs were then extracted using toluene and a Soxhlet extraction apparatus. A multi-stage chromatographic clean-up procedure removed potential chemical interferences and the extract was analyzed by Gas Chromatography - Tandem Mass Spectrometry (GC-MSMS). Further clean-up using carbon chromatography was used, as necessary, to remove interferences not removed by the open-column chromatographic clean-up.
All data were reported on an air dry solids basis.

The amounts of ^{13}C-labelled PCDD and PCDF surrogates that were recovered from each sample were used to determine percentage recovery. Those percentages were then used to correct the PCDD/F test results. The corrected PCDD/F concentrations were then reported for each sample.

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2.2.4 Data Analysis/Interpretation

Two different conventions were used to derive overall toxicity values for the soils and sewage biosolids included in the study. The present international convention (U.S. EPA, 1989) was used to calculate values expressed as International Toxicity Equivalents (I-TEQs), and an earlier convention was used to calculate values expressed as Homologue Toxicity Equivalents (H-TEQs). The latter values were derived to allow comparison with

Ontario soil background concentrations reported in the ***Ontario Typical Range of Chemical Parameters in Soil, Vegetation, Moss Bags and Snow*** (OMEE, 1994).

The I-TEQ for each soil and biosolid was derived by measuring the concentrations of all seventeen 2,3,7,8-chlorine substituted dioxin and furan congeners in the sample, multiplying each of the concentrations by the appropriate International Toxicity Equivalence Factor (I-TEF), and then summing the products. The I-TEF represents the toxicity of the congener relative to 2,3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD). The sum represents the total 2,3,7,8-TCDD toxicity equivalents (I-TEQ) for the sample.

Previous analytical methods measured only total concentrations of the five dioxin and five furan homologues, and the 2,3,7,8-TCDD congener. (Note: a homologue includes only congeners with the same number of chlorines). The H-TEQ for each soil and biosolid was derived by multiplying the homologue concentrations by the appropriate Toxicity Equivalence Factor (TEF), and summing the products. The sum represents the total 2,3,7,8-TCDD toxicity equivalents (H-TEQ) for the sample.

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The calculated I-TEQ values for the treated and non-treated soils were then entered into SYSTAT (ver. 6.1 for Windows), which was used to perform the Paired T-Test and the Sign Test. Budget constraints and high sample analysis cost limited the sample population to 10 paired sites, which was considered the minimum needed for statistical purposes.

2.3 Site Descriptions

Descriptions for the 10 paired sites are given in Table 2. Each description includes the general location of the site, and the following information for the treated and non-treated plots: 1) agricultural use (1996 crop, predominant crop); 2) soil type; and 3) surface texture.

The soil type was identified from the soil survey map for the area. The surface texture at each site was approximated using hand texturing techniques to estimate sand, silt and clay content. The locations of the paired sites are shown in Figure 2. Two sites near the Town of Hanover in Grey County occurred in close proximity to each other. Consequently, a single location is shown in Figure 2 for those sites.

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Field Survey of Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans (PCDD/Fs) in Sewage Biosolids Treated Agricultural Soils

3.0 Analytical Results

There are over 200 congeners of polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs). Analytical results are reported both as totals for the 10 different homologues (members of a homologue have the same number of chlorines), and as individual results for the 17 congeners that have substituted chlorines in the 2,3,7,8- positions on the molecule. The latter are the congeners of greatest concern with respect to toxicity.

The international toxicity values (I-TEQs) for the soils and sewage biosolids included in this study are presented in Table 3. Similarly, the homologue toxicity values (H-TEQs) for these materials are presented in Table 4. Data for congeners and homologue groupings in the soils are presented in Table 5, and in the sewage biosolids are presented in Tables 6 and Table 7.

The proportion of the different congeners and homologues present in a sample is a signature which can sometimes give clues to the origins of the dioxins and furans. For this reason, the percent occurrence of the different congeners and homologue groups is presented in Table 8.

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4.0 Interpretation of Analytical Results

4.1 General

When analyzing and interpreting the results from a study dealing with dioxin and furan analysis, the handling of data below the laboratory method detection limits (MDLs) can play an important role. For this study, it was decided that comparing the results to existing soil background information was important. Consequently, the same methods of handling MDLs were used that were employed in the development of the Ontario soil background concentrations for soils at uncontaminated rural parkland sites (OMEE, 1994). Thus, one half of the reported MDL for the congener or homologue for the particular sample was used for the I-TEQ and H-TEQ calculations. The values reported in Tables 5, Table 6 and Table 7, preceded by a less than (<) sign, are MDLs.

From Table 3, it can be seen that the mean I-TEQ concentrations for both the treated and non-treated soils is 1.9 ng/kg I-TEQ. A Paired T-Test indicated there was no difference between the two treatment means ($p = 0.54$). As a caution against the lack of a normal distribution of the data, a Sign Test was also conducted to determine if the number of occurrences of the treated soil having higher concentrations than the non-treated soil was significant. The Sign Test indicated that, although the treated soils had equal or higher values at seven of the ten sites, there was no difference between the two treatments ($p = 0.34$). For a sign test with 10 pairs to be significant, one treatment must have nine or more values higher than the other treatment. Thus, there was no significant increase in PCDD/F concentrations in the soils which received three applications of sewage biosolids in accordance with the sewage biosolids guidelines.

From Table 8, it can be seen that the congener profiles are quite different for the biosolids and soils. The sewage biosolids show considerably more occurrence of the T4CDDs, T4CDFs, and 2,3,7,8- substituted congeners of P5CDFs, P5CDDs, H6CDFs and H6CDDs, than do the soils. In addition, the biosolids have a lower occurrence of 1,2,3,4,6,7,8-H7CDF than the treated soils, but this may result from much higher detection levels of this substance in the biosolids than in the soils. MDLs for this congener in sewage biosolids varied considerably, and were up to 50 times higher than for other congeners or for the same congener in soils.

It can also be seen from Table 8 that, although two congeners show slightly higher occurrence in the treated soils than in the non-treated soils, the overall congener profile is very similar for both soil groups and there is no clear tendency for the treated soil profile to mimic that of the sewage biosolids. Since 2,3,7,8-TCDD is the congener of greatest concern from a toxicity perspective, it is of interest that it was not detected in any of the twenty soil samples or seven sewage biosolids samples.

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4.2 Comparison with Maximum Concentrations Allowed in Ontario

The mean PCDD/F concentration calculated for the treated soils was 1.9 ng/kg I-TEQ, and the individual concentrations ranged from 1.5 ng/kg I-TEQ in the Whitby, Ayr and Hanover #1 treated soils, to 3.5 ng/kg I-TEQ in the Oakville #2 treated soil (Table 3). In addition, the maximum concentration of PCDD/Fs determined for all soils (treated and non-treated) was 3.8 ng/kg I-TEQ, for the non-treated soil at the Oakville #2 paired site. These values are all well below the generic soil remediation concentration of 10

ng/kg I-TEQ for agricultural land use (OMEE, 1997). They are also well below the background soil remediation concentration of 7 ng/kg I-TEQ (OMEE, 1997), which is the upper limit of background PCDD/F concentrations in soils at uncontaminated rural parkland sites (OMEE, 1994).

The mean PCDD/F concentration calculated for the sewage biosolids was 10.8 ng/kg I-TEQ, and the individual concentrations ranged from 5.9 ng/kg I-TEQ in the Whitby sewage biosolids, to 17.8 ng/kg I-TEQ in the Halton sewage biosolids (Table 6). These values also are well below the interim maximum concentration of 100 ng/kg I-TEQ currently allowed in waste materials utilized on agricultural land (Pers. Comm., 1997).

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4.3 Comparison with Findings of Other Studies

The background PCDD/F concentrations derived for soils comprising uncontaminated rural parkland sites in Ontario (OMEE, 1994), were calculated using the earlier convention previously described in Section 2.2.4. Therefore, they can be directly compared to the homologue toxicity equivalent values (H-TEQs) presented in Table 4.

From Table 4, it can be seen that the mean H-TEQ for the non-treated soils is 1.3 ng/kg. This value is slightly lower than the mean background concentration of 1.7 ng/kg H-TEQ (OMEE, 1994). The highest value of 4.1 ng/kg H-TEQ determined for the non-treated soils at the Oakville #2 site, is slightly lower than the reported 98th percentile value of 4.8 ng/kg H-TEQ (OMEE, 1994), which indicates that 98% of the soils included in the background database have an H-TEQ value which is less than 4.8 ng/kg.

Again from Table 4, it can be seen that the mean H-TEQ value of 1.2 ng/kg for the sewage biosolids treated soils is slightly lower than for the non-treated soils. Further, the values for the treated soils at the Whitby, Oakville #1 and Oakville #2 paired sites were slightly lower than the values for the non-treated soils at those sites. These findings suggest that natural soil concentrations of PCDD/Fs are variable, and the range is such that natural concentrations may exceed treated concentrations in soils comprising the same landscape. When the treated and non-treated soil PCDD/F concentrations were compared to background soil concentrations, they were within the normal range reported (OMEE, 1994).

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From Tables 3 and Table 4, it can be seen that two sites have total toxicity values for both the treated and non-treated soils, that are approximately 2 times (I-TEQs, Table 3) and 4 times (H-TEQs, Table 4) those of the other sites. Both of these sites occur near Oakville, within the Greater Toronto Area. These findings suggest that other factors, such as high natural soil PCDD/F levels, or down-wind deposition from nearby urban centres, also influence soil PCDD/F concentrations. Indeed, a tendency towards higher concentrations of PCDD/Fs in soils located within urban areas has been noted by other researchers (Pearson et al., 1990).

When a large number of results are reported at below analytical method detection limits (MDLs), the method of handling the MDL data can have a significant influence on the results. Using the I-TEQ calculation method, if all the results for a particular sample were non-detects, and one-half of the MDL were used for calculation purposes, then the I-TEQ would be 1.4 ng/kg. The analogous H-TEQ would be 0.6 ng/kg.

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Of the 10 sites included in the study, only the two Oakville sites had I-TEQ values higher than 1.6 ng/kg (Table 3). Similarly, only the two Oakville sites had H-TEQ values which were markedly higher than 0.7 ng/kg (Table 4). When comparing the MDL based I-TEQ and H-TEQ values to those in Tables 3 and 4 respectively, it can be seen that the differences between the calculated toxicity values and those obtained using non-detect data, are negligible at most sites.

The mean concentration of PCDD/Fs of 10.8 ng/kg I-TEQ for the seven sewage biosolids examined compares favourably with those reported by Webber and Nichols (1995), and Webber and Bedford

(1996). In those studies, the authors examined the concentrations of organic chemical compounds in 16 Canadian sewage biosolids including 7 from Ontario. They found that mean PCDD/F concentrations varied from 4 ng/kg I-TEQ (Highland Creek sewage treatment plant, Toronto, Ontario), to 120 ng/kg I-TEQ (City of Edmonton, Alberta). The mean for the 16 sewage biosolids examined was 30 ng/kg I-TEQ, and the mean for the seven Ontario sewage biosolids was 10 ng/kg I-TEQ.

It has been reported that a significant exposure route of PCDD/Fs to humans may occur if livestock are allowed to graze on recently treated pastures and thereby ingest soil and sewage biosolids adhering to the plants (Wild et al., 1994). The current study did not examine concentrations of PCDD/Fs on plants growing in pastures where sewage biosolids had been recently applied. Hence, that route of exposure can not be discounted. For this reason, it is important that the waiting periods specified for hay and pasture in the sewage biosolids guidelines (OMEE and OMAFRA, 1996), be adhered to. They are designed to minimize direct ingestion of sewage biosolids by livestock.

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4.4 Theoretical Increase in Soil PCDD/F Concentration (I-TEQ) from Sewage Biosolids Applications

The treated soils received three applications of sewage biosolids under the conditions specified in three separate OME Part V approvals (Certificates of Approval). For aerobic biosolids, the sewage biosolids guidelines specify that the maximum amount of biosolids that can be applied over a 5 year period (which is the usual OME approval period), is 8 tonnes of solids per hectare, or 8,000 kilograms (kg) of solids per hectare (ha). The guidelines do not specify a maximum rate of application for anaerobic biosolids. They do, however, place limits on nitrogen application and metal additions to the receiving soils, which effectively preclude rates significantly higher than 8 tonnes of solids/ha under each OME approval.

If it is assumed that each treated soil had received approximately 24 tonnes (or 24,000 kg) of solids/ha under three OME approvals, this would theoretically have raised the PCDD/F concentration in the receiving soil by:

24,000 kg/ha x (10.8 ng/kg I-TEQ)a / (2,000,000 kg soil/ha)b = 0.13 ng/kg I-TEQ

a Mean PCDD/F concentration for sewage biosolids, from Table 3.
b Approximate weight of a hectare furrow slice.

The projected increase of 0.13 ng/kg I-TEQ in the receiving soil is only 6.8% of the mean of the non-treated soils. Further, the coefficient of variation (i.e. the variance/mean) for replicate samples in the Ontario background database is about 23% (OMEE, 1994). Therefore, this theoretical increase would result in a "treated" soil I-TEQ value which would be within the range of variability of PCDD/F levels in soils comprising uncontaminated rural parkland sites in Ontario (OMEE, 1994).

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5.0 Conclusions

The objectives of the study were attained with one minor exception. The sewage biosolids targeted for inclusion in the study were those produced by water pollution control plants (WPCPs) which had previously supplied all of the sewage biosolids applied on the treated soils. It was thought that this would provide a good indication of the total amount of PCDD/Fs which had been applied to the treated soils. It was found, however, that five of the treated soils included in the study had not received sewage biosolids from a single-source WPCP or regional storage facility. Rather, they had received sewage biosolids from two or more sources.

Measured and mean concentrations of PCDD/Fs were determined for: 1) ten Ontario agricultural soils which had received three applications of sewage biosolids; 2) ten adjacent or nearby agricultural soils which had not received sewage biosolids; and 3) seven sewage biosolids utilized on Ontario agricultural soils.

The concentrations of PCDD/Fs determined for the treated soils were compared to: 1) the generic soil remediation concentration of 10 ng/kg I-TEQ for agricultural land use (OMEE, 1997); 2) the background soil remediation concentration of 7 ng/kg I-TEQ for all land uses (OMEE, 1997); and 3) the concentrations reported in other studies. It was found that the ten I-TEQ concentrations for the treated soils were all well below the 7 ng/kg I-TEQ background concentration. They also fell within the normal range of background concentrations for soils comprising uncontaminated rural parkland sites (OMEE, 1994).

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Moreover, it was found that there was no significant difference in the concentrations of PCDD/Fs between the sewage biosolids treated and non-treated soils. Therefore, there was no evidence from this limited study that three applications of sewage biosolids in accordance with the sewage biosolids guidelines (OMEE and OMAF, 1996), increased the concentrations of PCDD/Fs in the receiving soils.

The concentrations of PCDD/Fs in the sewage biosolids were also compared to the interim maximum concentration of 100 ng/kg I-TEQ currently allowed in waste materials utilized on agricultural land (Pers. Comm., 1997). It was found that all of the sewage biosolids I-TEQ concentrations were well below the 100 ng/kg I-TEQ criterion.

In regard to individual congeners, the 2,3,7,8-TCDD congener is of greatest concern from a toxicity perspective. It was not detected in any of the twenty soils (treated and non-treated), or seven sewage biosolids sampled as part of the study.

Thus, based on the findings of this limited study, it is concluded that the application of sewage biosolids to agricultural soils, in accordance with the sewage biosolids guidelines (OMEE and OMAF, 1996), has not resulted in increased PCDD/F risk to human and animal health.

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Table 1. Number of Samples by Region/County

Region/County	Sewage Biosolid (Source)	Soil Samples	
		Treated	Non-treated
Regional Municipality of Durham	Whitby (WPCP)[1]	2	2
Regional Municipality of Halton	Halton (RSF)[2]	2	2
Regional Municipality of Niagara	Grimsby (WPCP)	1	1
Regional Municipality of Waterloo	Kitchener (RSF)	1	1
Bruce County	Southampton (WPCP)	1	1
Grey County	Owen Sound (WPCP)	1	1
	Hanover (WPCP)	2	2
TOTALS	**7**	**10**	**10**

[1] WPCP = Water Pollution Control Plant

[2] RSF = Regional Storage Facility; blend of sewage biosolids from more than one WPCP.

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Table 2. Descriptions of Paired Sites - Sewage Biosolids Treated and Non-treated Soils

Paired Site (Region or County)	Sewage Biosolid	Paired Sites							
		Treated Soil				Non-treated Soil			
		Agricultural Use		Soil Features		Agricultural Use		Soil Features	
		1996	Predominant	Soil Type	Soil Texture[1]	1996	Predominant	Soil Type	Soil Texture[1]
Ayr (Waterloo)	Kitchener	Corn	Corn, beans	Brady	SL	Corn	Corn, Beans, Wheat	Brady	SL
Smithville (Niagara)	Grimsby	Corn	Corn, beans	Peel	C	Corn	Corn, Beans	Peel	C
Southampton (Bruce County)	Southampton	Hay	Hay, cereal	Listowel	FSL	Pasture	Pasture	Listowel	FSL
Owen Sound (Grey County)	Owen Sound	Corn	Hay, corn	Harkaway	L	Pasture	Pasture	Harkaway	L
Hanover #1 (Grey County)	Hanover	Cereal	Hay, cereal	Sullivan	SL	Hay	Hay	Sullivan	SL
Hanover #2 (Grey County)	Hanover	Corn	Hay, corn	Burford	GL	Beans	Hay, cereal	Burford	GL
Whitby (Durham)	Whitby	Beans	Hay, cereal	Darlington	L	Cereal	Hay, cereal	Darlington	L
Port Perry (Durham)	Whitby	Cereal	Corn, cereal	Bondhead	VFSL	Alfalfa	Corn	Bondhead	VFSL
Oakville #1 (Halton)	Halton	Corn	Corn, beans	Oneida	CL	Clover	Fallow	Chinguacousy	CL
Oakville #2 (Halton)	Halton	Fallow	Corn, beans, cereal	Chinguacousy	L	Pasture	Pasture	Chinguacousy	L

Legend: GSL - Gravelly sandy loam; GL - Gravelly loam; FSL - Fine sandy loam; CL - Clay loam; VFSL - Very fine sandy loam; C - Clay; SL - Sandy loam; L - Loam

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Table 3. I-TEQs (ng/kg) for Sewage Biosolids and Soils

Site	Sewage Biosolids	Treated Soil	Non-Treated Soil
Smithville	11.5	1.6	1.5
Whitby	5.9	1.5	1.6
Port Perry		1.6	1.5
Ayr	13.5	1.5	1.5
Oakville #1	17.8	2.6	2.9
Oakville #2		3.5	3.8
Owen Sound	12.7	1.6	1.5
Southampton	8.2	1.6	1.5
Hanover #1	6.2	1.5	1.5
Hanover #2		1.6	1.5
Mean	**10.8**	**1.9**	**1.9**

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Table 4. H-TEQs (ng/kg) for Sewage Biosolids and Soils

Site	Sewage Biosolids	Treated Soil	Non-treated Soil
Smithville	15.6	0.9	0.8
Whitby	4.7	0.7	0.8
Port Perry		0.8	0.7
Ayr	12.5	0.7	0.7
Oakville #1	15.7	2.5	3.3
Oakville #2		3.6	4.1
Owen Sound	8.5	0.7	0.7
Southampton	6.4	0.9	0.8
Hanover #1	6.4	0.7	0.7
Hanover #2		0.8	0.7
Mean	**10.0**	**1.2**	**1.3**

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Table 5. Analytical Results (ng/kg) for PCDDs and PCDFs in Sewage Biosolids Treated and Non-Treated Soils

Homologue or Congener	I-TEF	Smithville		Whitby		Port Perry		Ayr		Oakville #1	
		Treated	Non-treated	Treated	Non-treated	Treated	Non-treated	Treated	Non-treated	Treated	Non-Treated
Homologue											
T4CDD total	1 (0)	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
2378-T4CDD	1 (1)	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
P5CDD total	0.5 (0.1)	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
H6CDD total	0.1 (0.3)	4.1	1.6	2.1	2.3	<1	<1	<1	<1	15	19
H7CDD total	0.01 (0.6)	15	9.6	6.9	12	12	5.2	7.5	6	76	95
O8CDD	0.001 (1)	58	30	20	56	62	17	28	16	260	320
T4CDF total	0.1 (0.1)	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
P5CDF total	0.55 (0.15)	<1	<1	<1	<1	<1	<1	<1	<1	1.6	4.1
H6CDF total	0.1 (0.05)	1.4	1.3	<1	<1	<1	<1	<1	<1	9.6	14
H7CDF total	0.01 (0.75)	4.4	3.7	1.8	4.1	5.6	<1	<1	<1	28	39
O8CDF	0.001 (1)	3.9	2.7	2.2	4.1	8.7	1.6	1.9	1.6	17	22
H-TEQ		0.9	0.8	0.7	0.8	0.8	0.7	0.7	0.7	2.5	3.3
Congener											
2378-T4CDD	1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
12378-P5CDD	0.5	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
123478-H6CDD	0.1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
123678-H6CDD	0.1	<1	<1	<1	<1	<1	<1	<1	<1	3.2	4.2
123789-H6CDD	0.1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
1234678-H7CDD	0.01	8.4	5.3	3.8	7.4	7	3.1	4.3	3.3	50	62
O8CDD	0.001	58	30	20	56	62	17	28	16	260	320
2378-T4CDF	0.1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
12378-P5CDF	0.05	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
23478-P5CDF	0.5	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
123478-H6CDF	0.1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
123678-H6CDF	0.1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
234678-H6CDF	0.1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
123789-H6CDF	0.1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
1234678-H7CDF	0.01	2.5	2.3	1.8	2.2	2.7	<1	<1	<1	10	14
1234789-H7CDF	0.01	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
O8CDF	0.001	3.9	2.7	2.2	4.1	8.7	1.6	1.9	1.6	17	22
I-TEQ		1.6	1.5	1.5	1.6	1.6	1.5	1.5	1.5	2.6	2.9

Notes: 1) Where result was < method detection limit, H-TEQ and I-TEQ calculations used 1/2 of method detection limit.

2) I-TEF: International Toxicity Equivalence Factor; numbers in brackets are proportionality factors used in H-TEQ calculations.

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Table 5: Analytical Results (ng/kg) for PCDDs and PCDFs in Sewage Biosolids Treated and Non-Treated Soils - cont'd.

Homologue or Congener	Oakville #2		Owen Sound		Southampton		Hanover #1		Hanover #2	
	Treated	Non-Treated	Treated	Non-Treated	Treated	Non-Treated	Treated	Non-Treated	Treated	Non-Treated
Homologue										
T4CDD total	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
2378-T4CDD	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
P5CDD total	1.6	1.8	<1	<1	<1	<1	<1	<1	<1	<1
H6CDD total	36	33	<1	1.4	3.8	2.2	<1	<1	1.9	<2
H7CDD total	130	150	11	9.8	16	11	12	11	19	3.7
O8CDD	390	720	40	33	53	35	38	31	67	18
T4CDF total	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
P5CDF total	4.6	6.1	<1	<1	<1	<1	<1	<1	<1	<1
H6CDF total	3.7	3.6	<1	<1	<1	<1	<1	<1	<1	<1
H7CDF total	24	27	4.8	1.4	4.4	<1	<1	<2	2.1	<1
O8CDF	16	23	<3	<2	<3	<2	<2	<2	4	<2
H-TEQ	3.6	4.1	0.7	0.7	0.9	0.8	0.7	0.7	0.8	0.7
Congener										
2378-T4CDD	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
12378-P5CDD	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
123478-H6CDD	1.8	<2	<1	<1	<1	<1	<1	<1	<1	<1
123678-H6CDD	3.8	3.6	<1	<1	<1	<1	<1	<1	<1	<1
123789-H6CDD	3.9	4.1	<1	<1	<1	<1	<1	<1	<1	<1
1234678-H7CDD	71	86	5.8	5.5	8.7	6.4	6.7	6.1	10	3.7
O8CDD	390	720	40	33	53	35	38	31	67	18
2378-T4CDF	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
12378-P5CDF	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
23478-P5CDF	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
123478-H6CDF	<2	<1	<1	<1	<1	<1	<1	<1	<1	<1
123678-H6CDF	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
234678-H6CDF	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
123789-H6CDF	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
1234678-H7CDF	9.1	8.7	2.6	<2	2.3	<1	<2	<2	<2	<2
1234789-H7CDF	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
O8CDF	16	23	<3	<2	<3	<2	<2	<2	4	<2
I-TEQ	3.5	3.8	1.6	1.5	1.6	1.5	1.5	1.5	1.6	1.5

Notes: 1) Where result was < method detection limit, H-TEQ and I-TEQ calculations used 1/2 of method detection limit.

2) I-TEF: International Toxicity Equivalence Factor; numbers in brackets are proportionality factors used in H-TEQ calculations.

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Field Survey of Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans (PCDD/Fs) in Sewage Biosolids Treated Agricultural Soils

Table 6. Analytical Results (ng/kg) for PCDDs and PCDFs in Sewage Biosolids

Homologue or Congener	Owen Sound	Hanover	Kitchener	Whitby	Halton	Southampton	Grimsby
Homologue							
T4CDD total	<2	9.1	8.7	4.1	4.1	6.8	<4
2378-T4CDD	<1	<1	<1	<1	<1	<1	<1
P5CDD total	11	13	17	9.7	22	11	110
H6CDD total	41	34	76	25	140	40	61
H7CDD total	270	190	540	180	720	234	460
O8CDD	1900	1000	3700	1300	3300	1098	2700
T4CDF total	22	2.2	8.4	3.9	7.4	5.7	1.1
P5CDF total	9.2	8.3	8	2.8	8.5	7.5	9.9
H6CDF total	18	17	17	3.7	12	13	18
H7CDF total	31	54	34	14	37	27	31
O8CDF	610	88	80	33	680	37	330
H-TEQ	8.5	6.4	12.5	4.7	15.7	6.4	15.6
Congener							
2378-T4CDD	<1	<1	<1	<1	<1	<1	<1
12378-P5CDD	to 3.2	2.4	2.9	1.8	2.5	3.6	3.3
123478-H6CDD	1.6	1.4	<2	<1	1.8	1.9	<2
123678-H6CDD	6.4	5.3	12	4.5	19	5.5	10
123789-H6CDD	9.2	3.7	13	4.6	22	5.7	7.1
1234678- H7CDD	150	100	300	100	450	114	260
O8CDD	1900	1000	3700	1300	3300	1098	2700
2378-T4CDF	12	2.2	3.7	3.9	4.5	3.1	<2
12378-P5CDF	3.4	<1	<1	<1	1.8	1.3	<1
23478-P5CDF	3.4	<1	<2	<1	2.2	2.3	<2
123478-H6CDF	9.3	2.3	4.5	2.2	8.8	2.9	5.1
123678-H6CDF	3.1	1.6	2.3	<1	2.6	1.9	<2
234678-H6CDF	2.1	1.9	3.7	1	<3	2.2	3
123789-H6CDF	<1	<1	<1	<1	<1	<2	<2
1234678-H7CDF	<50	25	<40	<20	<50	21	<40
1234789-H7CDF	6.7	1.4	<2	<1	5.6	<1	3.2
O8CDF	610	88	80	33	680	37	330
I-TEQ	12.7	6.2	13.5	5.9	17.8	8.2	11.5

Notes: 1) Where result was < method detection limit, H-TEQ and I-TEQ calculations used 1/2 of method detection limit.

2) Results for the Southampton sewage biosolids are means based on the analytical results obtained for 5 samples.

3) See I-TEFs and proportionality factors listed in Table 5.

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Table 7. Analytical Results and Mean PCDDs and PCDFs (ng/kg) for Southampton Sewage Biosolids

Homologue or Congener	PCDD/F Concentrations (ng/kg)					
	Sample #1	Sample #2	Sample 3#	Sample #4	Sample #5	Mean
Homologue						
T4CDD total	6.1	6.8	7.3	<6	7.9	**6.8**
2378-T4CDD	<1	<1	<2	<1	<1	**<1**
P5CDD total	9.8	5.1	17	8.1	15	**11.0**
H6CDD total	41	38	43	36	40	**40**
H7CDD total	220	220	250	230	250	**234**
O8CDD	1000	990	1200	1200	1100	**1098**
T4CDF total	4.9	5.7	5.2	6.6	5.9	**5.7**
P5CDF total	7.5	5.5	9.3	5.3	10	**7.5**
H6CDF total	11	14	15	11	14	**13**
H7CDF total	29	31	33	32	9.8	**27**
O8CDF	37	28	44	48	30	**37**
H-TEQ	6.0	5.7	7.7	5.9	6.7	**6.4**
Congener						
2378-T4CDD	<1	<1	<2	<1	<1	**<1**
12378-P5CDD	3.3	<4	3.4	3.8	3.3	**3.6**
123478-H6CDD	1.9	1.7	1.7	<2	<2	**1.9**
123678-H6CDD	5.3	5.1	5.7	5.6	5.8	**5.5**
123789-H6CDD	6.2	4.7	5.7	6	<6	**5.7**
1234678-H7CDD	110	110	120	110	120	**114**
O8CDD	1000	990	1200	1200	1100	**1098**
2378-T4CDF	3.4	3.1	3.2	3	2.8	**3.1**
12378-P5CDF	1.8	<1	1.4	<1	1.1	**1.3**
23478-P5CDF	3.1	<2	1.8	2.5	1.9	**2.3**
123478-H6CDF	3.2	2.8	2.8	3	2.9	**2.9**
123678-H6CDF	2.2	1	2.2	2.1	2	**1.9**
234678-H6CDF	<2	2.3	2.5	<2	2.1	**2.2**
123789-H6CDF	<2	<1	<2	<2	<2	**<2**
1234678-H7CDF	19	20	23	21	<20	**21**
1234789-H7CDF	<1	<1	<1	<1	<1	**<1**
O8CDF	37	28	44	48	30	**37**
I-TEQ	8.5	7.5	8.8	8.5	7.6	**8.2**

Notes: 1) Where result was < method detection limit, H-TEQ and I-TEQ calculations used 1/2 of method detection limit.

2) See I-TEFs and proportionality factors in Table 5.

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Table 8. Percentage (%) of Sewage Biosolids and Soils with PCDD/F Concentrations above Method Detection Limits

Homologue or Congener	Sewage Biosolids	Treated Soils	Non-treated Soils	All Soils
Homologue				
T4CDD total	73	0	0	0
P5CDD total	100	10	10	10
H6CDD total	100	60	60	60
H7CDD total	100	100	100	100
O8CDD	100	100	100	100
T4CDF total	100	0	0	0
P5CDF total	100	20	20	20
H6CDF total	100	30	30	30
H7CDF total	100	80	50	65
O8CDF	100	70	60	65
Congener				
2378-T4CDD	0	0	0	0
12378-P5CDD	91	0	0	0
123478-H6CDD	45	10	0	5
123678-H6CDD	100	20	20	20
123789-H6CDD	91	10	10	10
1234678-H7CDD	100	100	100	100
2378-T4CDF	91	0	0	0
12378-P5CDF	45	0	0	0
23478-P5CDF	55	0	0	0
123478-H6CDF	100	0	0	0
123678-H6CDF	82	0	0	0
234678-H6CDF	73	0	0	0
123789-H6CDF	0	0	0	0
1234678-H7CDF	45	70	40	55
1234789-H7CDF	34	0	0	0

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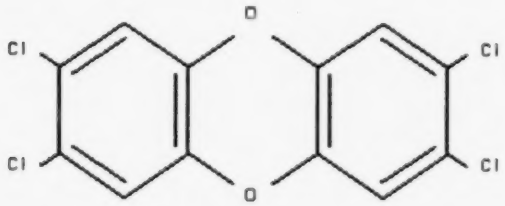
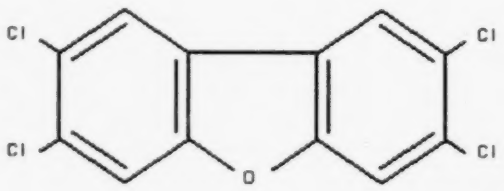
Field Survey of Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans (PCDD/Fs) in Sewage Biosolids Treated Ontario Agricultural Soils

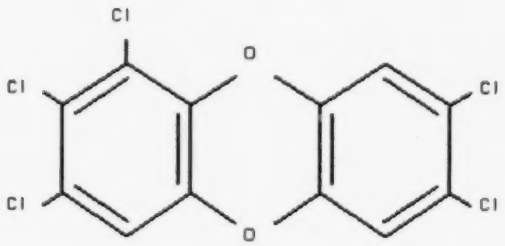
Figure 1. Molecular structures of some PCDD/Fs



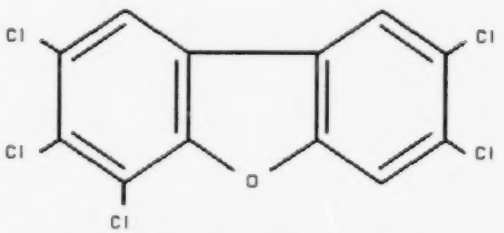
2,3,7,8-Tetrachlorodibenzo-p-dioxin



2,3,7,8-Tetrachlorodibenzofuran



1,2,3,7,8-Pentachlorodibenzo-p-dioxin



2,3,4,7,8-Pentachlorodibenzofuran

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Figure 2. Site Locations

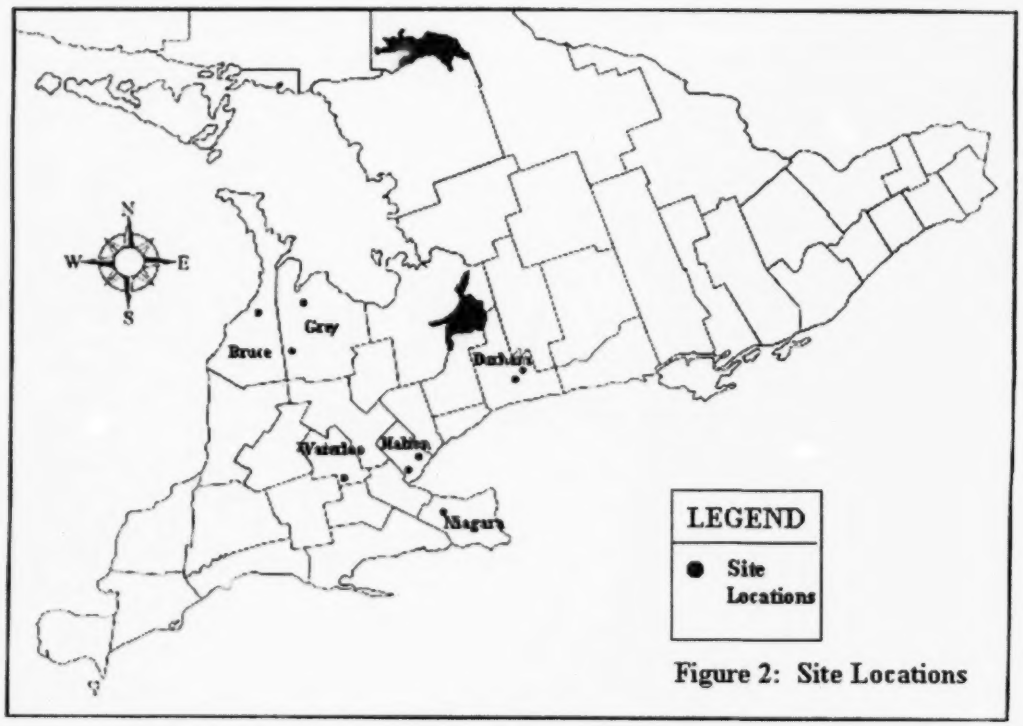

Figure 2: Site Locations

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